

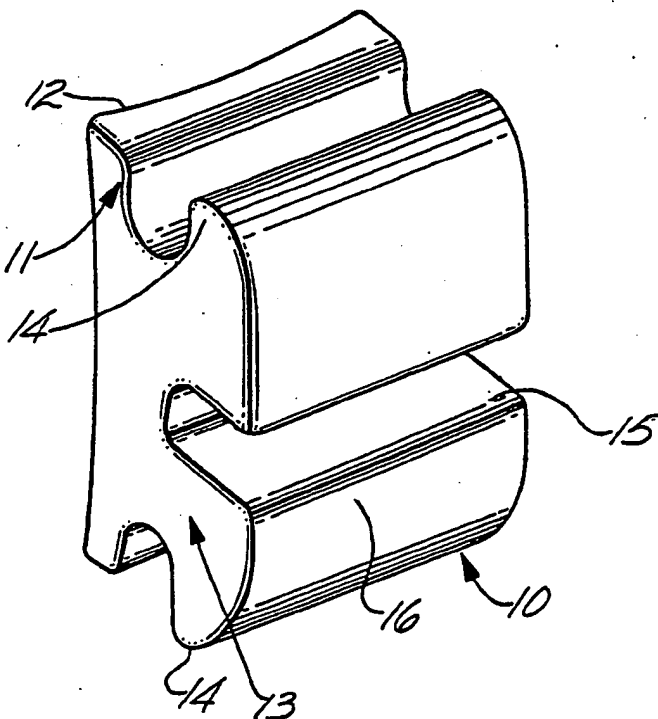


INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification⁴ : C04B 35/10, A61C 7/12 A61K 6/06	A1	(11) International Publication Number: WO 89/ 08085 (43) International Publication Date: 8 September 1989 (08.09.89)
(21) International Application Number: PCT/US88/00629 (22) International Filing Date: 2 March 1988 (02.03.88) (71) Applicant: UNITEK CORPORATION [US/US]; 2724 South Peck Road, Monrovia, CA 91016 (US). (72) Inventors: NEGRYCH, John, A. ; 15801 Los Lunas, Westminster, CA 92683 (US). GILLE, Henrick, K. ; 13760 Oxnard Avenue, Van Nuys, CA 91407 (US). KELLY, John, S. ; 10031 Lynrose Street, Temple City, CA 91780 (US). (74) Agents: SEIBEL, Richard, D. et al.; Christie, Parker & Hale, Post Office Box 7068, Pasadena, CA 91109-7068 (US).		(81) Designated States: AT (European patent), AU, BE (European patent), BR, CH (European patent), DE (European patent), FR (European patent), GB (European patent), IT (European patent), JP, LU (European patent), NL (European patent), SE (European patent). Published <i>With international search report.</i>

(54) Title: METHOD FOR MAKING CERAMIC ORTHODONTIC BRACKETS**(57) Abstract**

An orthodontic bracket or similar orthodontic appliance is made of a polycrystalline aluminum oxide having a translucency which minimizes visibility of the bracket when mounted on a tooth. The bracket is formed by pressing powdered high purity aluminum oxide plus sufficient magnesium oxide for controlling grain size, and sintering the resultant compact in hydrogen to yield a high density aluminum oxide bracket. The substantially color-free bracket has desirable strength and other mechanical properties combined with a translucency which permits the natural color of the tooth to show diffusely through in a fashion tending to make the bracket blend with and disappear against the tooth.



FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	FR	France	ML	Mali
AU	Australia	GA	Gabon	MR	Mauritania
BB	Barbados	GB	United Kingdom	MW	Malawi
BE	Belgium	HU	Hungary	NL	Netherlands
BG	Bulgaria	IT	Italy	NO	Norway
BJ	Benin	JP	Japan	RO	Romania
BR	Brazil	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KR	Republic of Korea	SE	Sweden
CG	Congo	LI	Liechtenstein	SN	Senegal
CH	Switzerland	LK	Sri Lanka	SU	Soviet Union
CM	Cameroon	LU	Luxembourg	TD	Chad
DE	Germany, Federal Republic of	MC	Monaco	TG	Togo
DK	Denmark	MG	Madagascar	US	United States of America
FI	Finland				

1

5

-1-

10

METHOD FOR MAKING CERAMIC ORTHODONTIC BRACKETS

Background of the Invention

15

Orthodontic treatment of improperly positioned teeth involves the application of mechanical forces to urge the teeth into correct alignment. The most common form of treatment uses orthodontic brackets which are small slotted bodies configured for direct cemented attachment to the front (labial) or rear (lingual) surfaces of the teeth, or alternatively for attachment to metal bands which are in turn cemented or otherwise secured around the teeth.

20

25

A resilient curved arch wire is then seated in the bracket slots, and the arch wire is bent or twisted before installation, whereby the restoring force exerted by the seated resilient wire tends to shift the teeth into orthodontically correct alignment. Depending on the shape of the arch wire, and the orientation of the bracket slot, it is possible to apply forces which will shift, rotate or tip the teeth in any desired direction.

30

35

Stainless steel is in many ways an ideal material for orthodontic brackets because this metal is strong, nonabsorbent, weldable, and relatively easy to form and machine. A significant drawback of metal appliances, however, relates to cosmetic appearance when the patient smiles. Adults and older children undergoing

-2-

1 orthodontic treatment are often embarrassed by the
"metallic smile" appearance of metal bands and brackets,
and this problem has led to various improvements in
recent years.

5 One relates to development of adhesives, bracket
bases, and techniques for direct cemented attachment of
brackets to at least the anterior teeth which are
prominently displayed when smiling. Direct cementation
eliminates the need for metal toothbands which are a
10 major factor in the metallic-smile problem. Part of
this has included development of smaller brackets which
are less obtrusive.

15 Still another area of improvement involves use of
nonmetal materials for the brackets. Plastic
orthodontic brackets have been used, but plastic is not
an ideal material because it lacks the structural
strength of metal, and is susceptible to staining and
other problems. Some of these problems are solved or
alleviated by ceramic materials which have recently been
20 proposed for orthodontic brackets. Both the plastic and
ceramic materials present a significantly improved
appearance in the mouth, and often the only visible
metal component is a thin arch wire which is
cosmetically acceptable. It has been proposed to use
25 single crystal sapphire for brackets, but transparent
ceramics have undesirable prismatic effects and single
crystal brackets are subject to cleavage. Other ceramic
brackets have been largely opaque so that they either do
not match tooth color or require coloring which is
30 uneconomic.

35 This invention is directed to a ceramic bracket
which achieves further cosmetic improvement by having a
translucent quality which takes on the color of the
underlying tooth to make the bracket blend with the
tooth. From the appearance standpoint, the translucent

-3-

1 bracket is a significant improvement over both
transparent and opaque brackets of nonmetallic
construction.

5 Summary of the Invention

The improvement of this invention relates to a
method for making a ceramic orthodontic bracket, by
pressing a powder consisting essentially of aluminum
oxide plus magnesium oxide in the range of from 0.05 to
10 0.3 percent by weight, at a sufficient pressure for
forming a compact having a shape corresponding to at
least a portion of the shape of the completed bracket,
and sintering the compact at a temperature in the range
of from 1750 to 1850°C for a sufficient time for forming
15 a bracket that is polycrystalline, has sufficient
strength for withstanding the loads applied during
orthodontic correction, and has sufficient translucency
that visible light emitted from the front surface of the
bracket comprises a portion backscattered from within
20 the bracket and a sufficient portion transmitted from
the base of the bracket to take on the color of an
underlying tooth.

Preferably the time and temperature of sintering
are such that the bracket has an in-line optical
25 transmittance for visible-light of at least 20% and
preferably in the range of from 20 to 60%. The body is
of a neutral color which, taken in combination with the
important property of translucency, makes the bracket
nearly invisible when viewed against the tooth from a
30 relatively short distance.

35

-4-

1 Description of the Drawing

 The drawing is a pictorial view of an orthodontic bracket made according to the invention.

5

10

15

20

25

30

35

-5-

1 Description of a Preferred Embodiment

 The drawing shows an exemplary orthodontic appliance in the form of an orthodontic bracket 10. The bracket has a base 11 suitable for either direct bonding to a tooth, or attachment to any kind of mounting fixture. A tooth-facing surface 12 of the base 11 is preferably conventionally concavely curved about both a mesiodistal axis and an apical axis to match the natural convexity of the tooth labial surface, but other curvatures can be used to accommodate lingual bracket positioning.

 A bracket body 13 extends from the base 11 to define bracket tie wings 14 for ligature anchorage, and a mesiodistally oriented arch-wire slot 15 extending from an outer body surface 16 into the bracket body. The presence or absence of tie wings (of either single- or twin-wing configuration) is not a feature of the invention, and the base and arch-wire slot may be angulated as desired to minimize or eliminate torquing or other bends of the arch wire.

 The orthodontic bracket is translucent since it is a polycrystalline article made of alpha aluminum oxide. It is important that the aluminum oxide has a high degree of optical transmittance in the visible spectrum, but also that it diffuse the light passing through the bracket. As is well known, human teeth have a broad range of color (quantified, for example, by the commercially available Vita shade system covering the range A1 through D4), and to make the improved orthodontic bracket effectively "disappear" when in place, it should assume the color of the underlying tooth. Thus, the ceramic material should be neutral, and neither add color to the light passing through nor subtract color by appreciable absorption. Aluminum oxide is particularly suitable since its optical

-6-

1 transmittance is substantially constant throughout the
visible spectrum and it therefore does not change the
color of light passing through the bracket. It is also
desirable since it is strong, hard, inexpensive and
5 readily available. The mechanical properties of
aluminum oxide can be distinguished from the relatively
low order properties available in organic materials such
as plastics which may also be translucent.

10 It has been proposed to use transparent single
crystal aluminum oxide or sapphire for orthodontic
brackets. This material is grown in the form of a
single crystal or closely aligned bicrystals having a
cross section close to the desired cross section of the
15 bracket. The crystal is grown in rods which are sliced
to the size of individual brackets. These can then be
cut and shaped to their final form by abrasive grinding.
The idea was that the highly transparent bracket would
show the tooth color. Such a transparent bracket also
has refractive effects and does not fully achieve the
20 desired result. Teeth are neither glassy nor opaque,
and a transparent bracket may still be quite noticeable.

More significantly, the single crystal material is
subject to cleavage under loads that occur in the course
of orthodontic treatment. Essentially point forces of
25 very high magnitude are applied to orthodontic brackets
by loading of the associated arch wire and tie wings,
and also during chewing. These high point loads can
initiate cleavage along crystallographic planes of the
sapphire, resulting in breakage. A significant
30 shortcoming of single crystal aluminum oxide is a
consequence of its manufacturing. Single crystals or
large bicrystals or the like may be grown to near net
shapes by a modified Czochralski method. However,
grinding may be required to form the base, the arch wire
35 slot or other surfaces. The grinding introduces surface

-7-

1 imperfections which can have a devastating effect on
strength. Cracks initiating at such imperfections
propagate through the crystal, resulting in breakage at
abnormally low stresses, well below the stresses one
5 would expect.

It is desirable that the bracket be translucent
rather than transparent. Light passes through a
transparent ceramic in a straight line. Thus, when a
single crystal of sapphire is placed on a printed page,
10 the text can be read through the crystal. In a
translucent material, a large proportion of light passes
through the crystal, but not in a straight path.
Optical irregularities in the bulk material cause the
light passing therethrough to be refracted, reflected,
15 and otherwise scattered so that it is diffuse.

Translucence is a relative property of a material.
This can be visualized by considering water to which
milk is added. When a few drops of milk are added to
the water, it becomes cloudy or milky. The formerly
20 completely transparent water is now somewhat translucent
in that a portion of the light transmitted through the
solution is diffused by scattering from the milk
particles. As more milk is added, more of the light is
diffused until it becomes impossible to read through the
25 solution. Further, the solution takes on the color of
the milk as more light entering the front of the
solution is backscattered by the milk particles and less
is reflected from whatever surface is behind the
solution. When the solution is slightly cloudy an
30 overwhelming proportion of the light emitted from the
face of the solution is reflected from the surfaces
behind the solution and a minor proportion is
backscattered by particles of milk within the solution.
These proportions reverse as more milk is added.

35 It is significant that the translucence be a bulk

-8-

1 property of the material rather than a surface effect.
Some light diffusion can be obtained by roughening a
surface as, for example, with frosted glass. This is
not completely satisfactory in an orthodontic bracket,
5 however, since the surface is continually wet, and the
principal change in the index of refraction occurs at
the air-liquid interface which is nearly smooth.
Further, it is undesirable to have roughened surfaces on
orthodontic appliances because of the adhesion of
10 substances in the mouth. As pointed out above, rough
surfaces may also have imperfections which serve as a
source for initiation of cracks. Since ceramics do not
have the ductility of metals, roughness can
significantly degrade strength.

15 To minimize the contrast between the bracket and
the tooth, it should have the same color as the tooth.
Color is perceived due to light reflected or emitted
from a surface. One could form a spectrum of appliances
to match the range of natural tooth colors, but the cost
20 and inconvenience would be undesirable. It is better to
see the tooth color itself, as seen through a
translucent bracket.

In order for the orthodontic bracket to assume the
color of the underlying tooth, it is important that
25 sufficient light seen from the front surface of the
bracket attached to the tooth be light that has been
transmitted from the tooth surface, and that the tooth
color is not overwhelmed by light backscattered from
optical irregularities within the bracket. In other
30 words, a substantial amount of the incident light should
pass through the bracket, albeit diffused, to the base
for reflection off of the tooth surface, and then be
retransmitted through the bracket to be emitted from the
front surface.

35 Since the bracket is translucent rather than trans-

-9-

1 parent, a portion of the light is backscattered by the
internal optical irregularities in the bracket. The
backscattering is preferably minimized since the back-
scattered light tends to be white and will almost
5 invariably be different from the tooth color. Further,
by using a translucent aluminum oxide bracket, all of the
optical properties of the tooth are mimicked. Teeth are
not opaque and considerable attention has been devoted to
achieving limited translucence in materials used for
10 prostheses to mimic the replaced or repaired tooth. Such
concern is alleviated by a translucent bracket since
light transmitted through the tooth as well as that
light reflected from the front, is, in turn, emitted
substantially unchanged from the translucent bracket.

15 The amount of visible light transmitted through the
polycrystalline aluminum oxide used to make the bracket
is at least 20% and preferably is in the range of from
20% to 60%, and the light backscattered from internal
optical irregularities within the bracket is in the
20 range of from 40% to 80%. This translucence is measured
by in-line transmission of light through a specimen 0.5
mm thick, the light being in the wavelength range of
from 0.4 to 0.8 microns. This translucence assures that
the light seen from the front surface includes sufficient
25 light that has been reflected from the tooth surface to
take on the color of the underlying tooth.

The translucence measurement is made by illuminating
a sample 0.5 mm. thick with a collimated beam and
measuring the proportion of light emitted at the
30 opposite surface of the sample in the direction of the
collimated beam. Since the light is scattered by the
optical irregularities within the sample, a small
proportion may be transmitted in the direction of the
incident beam and a large proportion scattered in other
35 directions. This is to be distinguished from a

SUBSTITUTE SHEET

-10-

1 transmittance measurement where much of light is
absorbed by the medium through which it passes. The
scattering is desirable in the orthodontic bracket since
it conveys the color of the underlying tooth and
5 diffuses it without prismatic effects. Aluminum oxide
has little absorption and the limited absorption is
uniform throughout the visible spectrum so that no color
change is introduced.

10 In a preferred embodiment, translucence is obtained
in an orthodontic bracket by forming it from poly-
crystalline aluminum oxide which is inherently
transparent. By polycrystalline is meant that the
bracket is made of a ceramic having a multiplicity of
randomly oriented crystals self-bonded together. That
15 is, the adjacent crystals are separated by a grain
boundary of substantially the same material as the
crystals, rather than being cemented together by a
different material.

20 The orthodontic bracket is made by pressing and
sintering aluminum oxide. The parts are fabricated by
pressing powder to a desired shape and sintering the
pressed compact at a sufficient time and temperature
that the bracket is translucent and has sufficient
strength to withstand the high loads applied to the
25 bracket during orthodontic corrections.

The polycrystalline aluminum oxide has essentially
a single phase and substantially zero porosity to
maintain a high degree of optical transmittance. It is
preferably made of 99.99% alpha aluminum oxide to which
30 a small amount of magnesium oxide is added for
controlling grain growth. Starting with 99.99% aluminum
oxide is desirable for maximum strength and freedom from
chromatic effects. Magnesium oxide in the range of from
0.05% to 0.3% is added to the aluminum oxide. This
35 material limits grain growth so that the grain size of

SUBSTITUTE SHEET

-11-

1 the sintered polycrystalline bracket is not too large.
It is believed that a portion of the magnesium oxide
combines with the aluminum oxide in a spinel type
structure in the grain boundaries.

5 Thus, after sintering, the polycrystalline bracket
has an average grain size in the range of from 10 to 50
microns. The composition and the sintering time and
temperature should be controlled so that the average
10 grain size in the completed orthodontic bracket is in
this range for high optical transmittance and strength.
Preferably the average grain size is 30 microns and the
range of grain size is from 8 to 85 microns, which gives
good packing and high strength. If the grain size is
15 too large, strength may be reduced due to the greater
distances through which cracks may propagate before
encountering a grain boundary.

The particle size of the powder from which the
bracket is made is preferably in the range of from 1/2
to one micron. The aluminum oxide powder is typically
20 chemically precipitated material and has reasonably
uniform grain size. Few particles as large as three
microns are found, and these may be agglomerates of
smaller particles.

To give the powder some green strength when
25 pressed into a compact, a small amount of a temporary
organic binder such as a paraffin wax, polyethylene
glycol or polyvinyl alcohol is included in the powder
mix. From 0.75 to 3% by weight of polyvinyl alcohol is
preferred. The polyvinyl alcohol is mixed with water to
30 wet the particle surfaces and the slurry is then spray
dried. The temporary binder is removed in subsequent
firing.

A measured quantity of the powdery mixture of
aluminum oxide powder, magnesium oxide powder and
35 temporary binder is placed in the die cavity of a high-

-12-

1 pressure hydraulic press. The amount of powder is just
enough to form one blank from which a bracket is formed.
The die has a cavity with a cross section corresponding
to at least a portion of the desired shape of the
5 appliance being formed. The arch-wire slot and the
undercuts under the tie wings may be completely or
partially formed in this operation, or are preferably
ground later.

A punch having the cross section of the die cavity
10 is pressed into the powder in the cavity at 1400 to 1550
kg/cm₂ to tightly pack the powder. In a preferred
embodiment, a lateral slide is also employed for forming
the curved base of the bracket. Such punches, dies, and
slides are conventionally used for pressing a broad
15 variety of metals or ceramics to desired shapes. After
pressing the powder, a green compact having at least
part of the shape of the finished bracket, albeit larger
because of subsequent shrinkage, is ejected from the die
cavity. Preferably, multiple die cavities are used in
20 commercial operations for high productivity.

Alternatively, the compact from which the bracket
is made may be extruded under pressure, formed by
injection molding, or may be compressed isostatically,
as are well known.

25 The somewhat fragile green compact is then fired in
air at a temperature in the range of from 1300 to 1400°C
for an hour. By heating in an oxidizing environment,
the organic binder and any inadvertent organic
contamination is vaporized or oxidized so as to be
30 removed completely and not affect the color of the
finished bracket.

The compact is slowly heated to the firing
temperature so that any residual carrier liquid,
vaporized binder and combustion products of the binder
35 can be removed without disrupting the delicate compact.

SUBSTITUTE SHEET

-13-

1 A heating rate of up to 100°C per hour has been found
satisfactory. Further, by heating to a temperature in
the range of from 1300 to 1400°C, some sintering of the
compact occurs, greatly increasing its strength for
5 subsequent handling.

The compact is then sintered at temperatures in the
range of from 1750°C to 1850°C in an environment of
flowing high purity hydrogen. The highest commercially
available purity of hydrogen is used.

10 The sintering time is preferably at least one hour,
somewhat longer times being preferred for lower tempera-
tures. The time should be at least sufficient to
produce a translucent bracket having an in-line
transmittance of visible light in excess of 20%. The
15 heating in hydrogen should be slow enough that hydrogen
replaces other gases in the interstices of the compact
before sintering closes paths through which gas can
escape. This helps eliminate residual porosity after
sintering.

20 In a preferred embodiment the bracket is maintained
at a temperature above 1750°C for at least five hours
and at the maximum sintering temperature for at least
one hour. Preferably the bracket is maintained above
1750°C for at least twelve hours and at the maximum
25 temperature for as much as six hours. The maximum
sintering temperature differs slightly for different
batches of raw materials and can be determined
empirically for obtaining the desired degree of
translucency and mechanical strength. For example, a
30 maximum sintering temperature of 1820°C for six hours
has been found suitable for most purposes. At such
sintering times and temperatures, the original particles
of aluminum oxide powder sinter together to form a
bracket having a density very close to 100% of the
35 theoretical density of alpha aluminum oxide.

-14-

1 For example, a translucent ceramic bracket is made
by mixing 99.99% aluminum oxide powder having an average
particle size of a little less than one micron with 0.2%
5 by weight of magnesium oxide powder. One percent by
weight of polyvinyl alcohol is added in sufficient
dionized water solution to make a slurry that can be
thoroughly mixed. The powder mixture is spray dried and
a measured quantity sufficient to make the blank for one
10 bracket is added to the cavity of a forming die. A
hydraulic press presses a punch into the die cavity at a
pressure of 1450 kg/cm₂ to form a green compact. The
green compact is then heated in air at a rate of 100°C
per hour to a maximum temperature of 1300°C where it is
15 held for an hour and then furnace cooled. The compact
is then heated in hydrogen to 1750°C at a rate of about
70°C per hour. It is slowly heated to 1820°C over a
period of about six hours, held at 1820°C for six hours,
and cooled over a period of about six hours to 1750°C.
20 The total cycle time from room temperature to room
temperature is in excess of 48 hours.

 After sintering, the brackets are tumbled in
conventional abrasive for slightly rounding the edges
and removing any undesirable "flash" or protrusions from
the surfaces. Finally, any machining operations are
25 conducted for shaping the completed bracket. For
example, the arch-wire slot and tie wing undercuts may
be ground with diamond wheels. The brackets may be
tumbled after grinding.

 The sintered polycrystalline aluminum oxide ortho-
30 donic bracket is translucent. The in-line optical
transmittance through the polycrystalline alumina is at
least 20%, and preferably is in the range of from 20 to
60%. Light passing through the bracket is, however,
diffused by the translucent polycrystalline aluminum
35 oxide.

-15-

1 The reason light passing through the
polycrystalline aluminum oxide is diffused and partly
backscattered is not completely known. Since the
material is polycrystalline, adjacent crystals have
5 different, largely random crystallographic orientations.
This results in variations in index of refraction along
any straight-line path through the bracket. Small
refractive effects may occur at grain boundaries,
resulting in a multiplicity of internal scattering
10 locations. The grain boundaries are sites of
crystallographic imperfections and these arrays of
imperfections may also have different indexes of
refraction which deflect light in a multiplicity of
directions.

15 Further, even though the aluminum oxide after
sintering has substantially zero porosity, traces of
residual porosity may remain in grain boundaries or
other locations in the finished product. Such traces of
porosity would have a pronounced effect on light
20 transmission, with resultant scattering and diffusion of
light passing through the polycrystalline material. It
is probable that a combination of these effects is
involved in producing the desired degree of translucence
in a pressed and sintered aluminum oxide orthodontic
25 bracket.

 It is significant that the bracket has a high
degree of optical transmittance, which is believed due
to the self bonding of high purity aluminum oxide in the
polycrystalline material. It has been proposed in the
30 past to form ceramic orthodontic brackets by pressing
and cementing aluminum oxide powder. For example, in
U.S. Patent No. 4,219,617 by Wallshein, aluminum oxide
powder is commingled with other ceramic materials having
a lower melting point than the aluminum oxide. The
35 mixed powders are pressed in a hydraulic press and the

-16-

1 resultant green compacts are sintered at temperatures in
the range of from 1575 to 1675°C, or about the melting
temperature of the other ceramic phase. The resultant
liquid bonds the aluminum oxide particles together,
5 forming a relatively dense and strong ceramic. This
liquid phase bonding is often referred to in the jargon
as "sintering," whereas it is more properly referred to
as "liquid phase sintering" or "cementing", depending on
the proportion of liquid present, since the individual
10 aluminum oxide particles are cemented together by a
second ceramic phase.

As a result of such processing, as described and
claimed by Wallshein, an orthodontic bracket is white or
slightly off-white. It may not be opaque since the
15 inherent transparency of ceramic materials used in the
manufacture will commonly let some light be transmitted
through such a bracket. Liquid phase cementing may
occur in porcelains, for example, and they have a slight
degree of translucence. The degree of optical transmit-
20 tance is, however, quite low and most of the light seen
is reflected from the surface in view. This results in
a milky white appearance where the ceramic has its own
"color," albeit white or off-white. Such color cannot,
of course, match the range of colors in human teeth, and
25 as Wallshein states, the composition may be colored to
the desired shade of white with a pigment to match
adjacent teeth. The cost of having an inventory of
orthodontic brackets to match the color range in teeth
is prohibitive.

30 A ceramic orthodontic bracket is secured to a tooth
with an adhesive substance. Good bonding of the
adhesive to the base of the bracket is important so that
it can withstand high occlusal forces and the loads
applied during orthodontic correction. Controlled
35 roughness of the base of the bracket may, therefore, be

-17-

1 desirable to enhance bonding strength of the adhesive to
the bracket. Preferably a smooth surface is provided on
the base, and the adhesive is securely bonded to a
5 prepared surface on the base. The base surface can be
prepared by depositing a glass frit on the base and
firing the frit for bonding the glass to the aluminum
oxide. The surface is then primed with an organo-silane
before application of the adhesive.

10 Surfaces of the orthodontic bracket should be
smooth. Smoothness is promoted by employing polished
dies and punches in the pressing operation. If desired,
the surfaces may be smoothed by grinding, ultrasonic or
abrasive polishing after sintering, however, that has
15 not proved necessary. A surface having a roughness of
about one half to one micron RMS is preferred.
Roughness is largely a function of grain size in a well
made bracket.

20 The pressing and sintering technique for forming a
polycrystalline aluminum oxide article from aluminum
oxide powder can result in an orthodontic bracket with
precise dimensions. Precision is enhanced by careful
control of the pressing operation for forming green
compacts and the mix of particle sizes in the aluminum
oxide powder. The sintering operation inherently causes
25 shrinkage from the green compact to the finished
article. The proportion of shrinkage can be controlled
by attention to powder quantity and quality, mold
geometry and pressure in green compact pressing. Care
in these techniques can produce finished orthodontic
30 brackets well within acceptable tolerance limits.

-18-

1 WHAT IS CLAIMED IS:

1. A method for making a translucent polycrystalline ceramic orthodontic bracket
5 characterized by:

pressing a powder consisting essentially of aluminum oxide plus magnesium oxide in the range of from 0.05 to 0.3 percent by weight at a sufficient pressure for forming a compact having a shape corresponding to at
10 least a portion of the shape of the completed bracket; and

sintering the compact at a temperature in the range of from 1750 to 1850°C for a sufficient time for forming a bracket that is polycrystalline, has sufficient
15 strength for withstanding the loads applied during orthodontic correction, and has sufficient translucency that visible light emitted from the front surface of the bracket comprises a portion backscattered from within the bracket and a sufficient portion transmitted from
20 the base of the bracket to take on the color of an underlying tooth.

2. A method as recited in claim 1 characterized by sintering the compact at a sufficient temperature and
25 for a sufficient time that the in-line transmittance of the bracket is at least 20% per 0.5 millimeter thickness.

3. A method as recited in claim 2 characterized by sintering the compact at a sufficient temperature and
30 for a sufficient time that the in-line transmittance of the bracket is in the range of from 20% to 60% per 0.5 millimeter thickness.

35

-19-

1 4. A method as recited in claim 1 characterized by
sintering the compact at a sufficient temperature and
for a sufficient time that the average grain size is in
the range of from ten to fifty microns.

5 5. A method as recited in claim 4 characterized by
sintering the compact at a sufficient temperature and
for a sufficient time that the average grain size is 30
microns.

10 6. A method as recited in any of the preceding
claims characterized by sintering the compact for at
least five hours at a temperature above 1750°C.

15 7. A method as recited in any of the preceding
claims characterized by sintering the compact at the
maximum sintering temperature for at least an hour.

20 8. A method as recited in any of the preceding
claims characterized by sintering the compact in a
hydrogen environment.

25 9. A method as recited in any of the preceding
claims characterized by the aluminum oxide particles
being sub-micron in size.

30 10. A method as recited in any of the preceding
claims characterized by the aluminum oxide particles
having an average particle size in the range of from 1/2
to one micron.

35 11. A method as recited in any of the preceding
claims wherein the powder also comprises a temporary
organic binder.

-20-

1 12. A method as recited in any of the preceding
claims characterized by the aluminum oxide powder
consisting essentially of 99.99% aluminum oxide.

5 13. A method as recited in any of the preceding
claims characterized by sintering the compact at a
sufficient temperature and time for reducing the
porosity of the bracket to substantially zero.

10 14. A method as recited in any of the preceding
claims characterized by preheating the compact in an
oxidizing environment at a temperature in the range of
from 1300 to 1400°C for at least one hour before
sintering.

15 15. A method as recited in claim 14 characterized
by heating the compact to the preheating temperature at
a maximum rate of 100°C per hour.

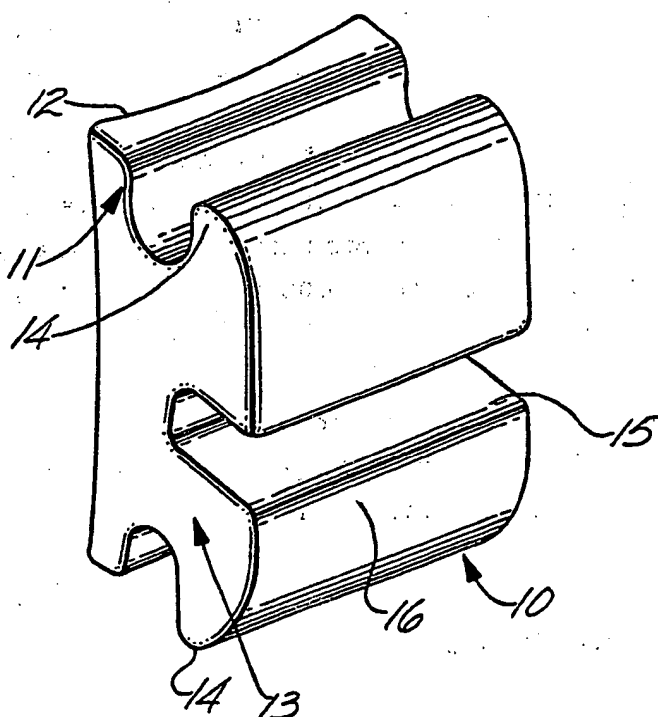
20 16. A method as described in the preceding
specification.

25

30

35

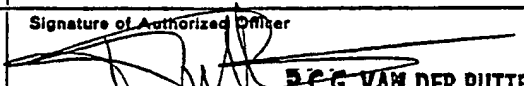
1/1



INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 88/00629

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁴		
According to International Patent Classification (IPC) or to both National Classification and IPC		
IPC ⁴ : C 04 B 35/10; A 61 C 7/12; A 61 K 6/06		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
IPC ⁴	C 04 B; A 61 C	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹		
Category ⁹	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
X	DE, A, 2120802 (FELDMUHLE ANLAGEN- UND PRODUKTIONSGESELLSCHAFT mbH) 9 November 1972 see claims 1,4,6; page 3, paragraph 4; page 4, paragraphs 1,2; page 6, paragraph 3 - page 7, paragraph 1	1-3,7,9, 11,13
A	--	4-6,8,10, 12,14-16
X	US, A, 4396595 (H.R. HEYTMETIJER et al.) 2 August 1983 see claims 1,3,13,14; column 3, lines 38-68; column 5, lines 1-20; column 7, lines 1-44	1,4-12
A	--	2,3,13-16
X	EP, A, 0218279 (N.V. PHILIPS' GLOEILAMPEN-FABRIEKEN) 15 April 1987 see claims 1-3; page 3, line 13 - page 4, line 1; page 5, lines 1-22	1,4-8,12, 14
A	--	2,3,9-11, 13,15,16
	--	./.
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>¹⁰ Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"A" document member of the same patent family</p> </div> </div>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
19th October 1988	10. 11. 88	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	 P.C.G. VAN DER PUTTEN	

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
X	US, A, 4219617 (M. WALLSHEIM) 26 August 1980 see claims 1, 12-14, 28, 29; column 6, lines 41-48; column 7, lines 3-23 -----	1-16

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO.**

US 8800629

SA 21561

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 03/11/88
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
DE-A- 2120802	09-11-72	None	
US-A- 4396595	02-08-83	JP-A- 58145661	30-08-83
EP-A- 0218279	15-04-87	JP-A- 62059569	16-03-87
		NL-A- 8502457	01-04-87
		US-A- 4699774	13-10-87
US-A- 4219617	26-08-80	None	